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CAPILLARY SUCTION STUDY OF THE SURFACE HOMOGENEITY OF SEMIFINISHED CERAMIC PRODUCTS AND ARTICLES

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The surface homogeneity of semifinished products and ceramic articles was investigated by capillary suction. The degree of surface inhomogeneity, determined by the rate of suction of a liquid by different sections of samples of different shape, fabricated by plastic molding, slip casting, and semidry molding, was compared. The effect of the firing temperature on the degree of surface inhomogeneity of the samples was established. The method is proposed for use for evaluating water suction of ceramics.

In the manufacture of quality ceramic articles, molding operations are determining [1]. Selection of the molding method is a function of the type of article and the process possibilities. For most articles, the semifinished product must have minimal, uniformly distributed porosity over the entire bulk, which ensures good strength properties.

There are nondestructive control methods for determining the homogeneity of mixtures, semifinished products, and samples, based on complex instrumental methods, for example, x-ray or γ-radiation [2, 3].

The dependence of the degree of inhomogeneity of samples molded by different methods, calculated with the concentration of markers in different parts of the semifinished product, on the product of a geometric factor (ratio of the sample volume to the area of application of molding force) and a molding factor (ratio of the maximum molding pressure to the relative viscosity of the system) was established in [4]. It was found that the geometric factor has the decisive effect on the inhomogeneity of the articles.

The surface structure of the semifinished product molded by any method differs from the structure of the inner sections and carries information on the molding regimes (methods of applying pressure in molding, rate of paste build-up in slip casting, etc.). By studying the surface porosity of an article, we can judge the homogeneity of samples obtained by any molding method. The surface state, in particular, its open porosity, is important for application of coatings — glazing and metal coating.

The porosity of ceramic semifinished products and articles can be investigated by different methods. There are methods of thermoporometry, mercury, water-air, water, and gas-liquid porometry, as well as complex methods [5 – 10]. These methods allow determining the overall pore structure of samples but do not give any information on the homogeneity of the surface or bulk of the sample.

There is a capillary method of nondestructive control based on capillary penetration of an indicator liquid inside a defect and designed to reveal defects opening on the surface of the object controlled (GOST 18442–80). This method is suitable for revealing cracks and pores with a cross section of 0.1 – 500 μm, including through pores, on the surface of ferrous and nonferrous metals, alloys, ceramics, glass, etc. The similar fuchsine method is widely used in porcelain-faience factories for revealing the cracks that arise in drying.

Data are reported in [11] on the correlation of the rate of suction of a liquid by the surface of a ceramic with its physical properties and microstructure. The method of investigation used is based on determining the coefficient of suction of a liquid by the surface of a ceramic sample, determined from a graph (Fig. 1). It was found that the suction coefficient is directly proportional to the fraction of open pores that actively absorb the liquid.

We modified the method used in [11] to obtain information on the homogeneity of the surface of individual sections of the semifinished product and the ceramic sample. Capillaries with the liquid (distilled water dyed with a solution of brilliant green) were brought into contact with the center and edge of three sides of the sample (top, bottom, and side surfaces). The amount of liquid absorbed was measured every 3 min for 15 min and the suction coefficients were then determined.

Porcelain and faience pastes and porcelain paste with a filler (0.6 mm porcelain grains) were used as the initial mate-

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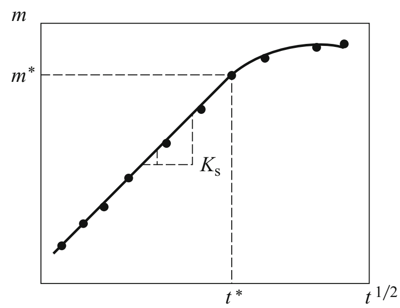

Fig. 1. Amount of absorbed liquid m as a function of the root of the suction time $t^{1/2}$: K_s) suction coefficient.

rials for preparing the samples. Samples of different shape (slabs, cylinders, and cylinders with a semicircular recess from the side of one of its ends) were molded by semidry molding, plastic molding, and casting in gypsum molds (Fig. 2). The overall f_{ov} and reduced f_{re} shape factors (see Table 1) were calculated for each sample shape:

$$F_{ov} = V/F_{ov};$$

$$F_{re} = V/F_{con},$$

where V is the volume of the sample; F_{ov} is the overall area; F_{con} is the area of the sample to which the molding force is applied.

The samples made from the porcelain and faience pastes in the form of slabs of different density were obtained by molding at different pressures. An almost linear dependence between the porosity of the semifinished product and the open porosity of the fired samples and suction coefficient K_s was established (Fig. 3). After firing at 1200°C, the K_s of the porcelain samples, despite their smaller open porosity, had a slightly higher value than the K_s of the faience samples. This can be attributed to the presence of a large number of small pores in the porcelain samples, for which the suction rate of the unit of surface area is higher than for pores of much larger radius but smaller number (Fig. 4).

The inhomogeneity coefficient was introduced to assess the degree of inhomogeneity of the surface of the samples; it was determined with the ratio of the difference of the suction coefficients to the difference of the distances between suction points:

$$H = dK_s/dx,$$

where dK_s is the difference of the suction coefficients of the

TABLE 1

Sample shape	Average (overall/reduced) shape factor	Overall/reduced shape factor of samples formed by		
		plastic molding	semidry molding	casting in gypsum molds
Cylinder	4.8/16.0	5.0/22.0	4.8/10.0	4.5/6.0
Slabs	2.9/7.0	3.0/9.0	2.7/4.0	3.0/4.1
Cylinder with recess	2.2/5.8	2.1/7.2	2.1/3.6	2.5/3.1

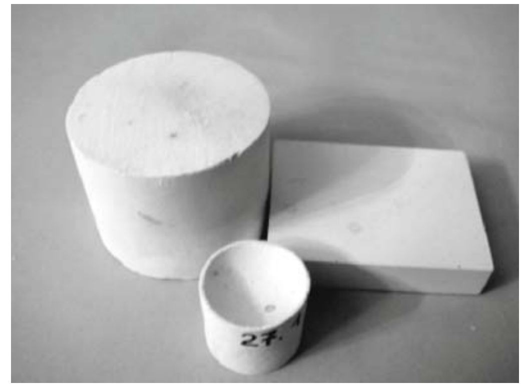

Fig. 2. Samples studied.

liquid between two neighboring points on the corresponding side; dx is the distance between these points.

The maximum value was used as the inhomogeneity coefficient of the sample.

A dilatometric analysis was conducted to select the characteristic firing temperatures at which the structure of the samples changed most markedly. The analysis showed that at temperatures above 950°C, both for porcelain and for faience samples, sintering begins and then actively takes place. Sintering takes place most intensively up to 1200°C. The samples in each batch were fired at 1000, 1100, and 1200°C and held at the maximum temperature for 1 h.

In comparing the homogeneity of the surface of semifinished products and fired samples, the samples in the shape

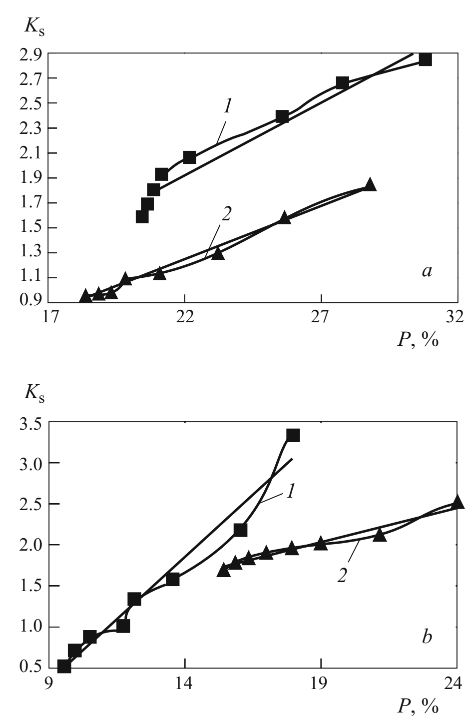

Fig. 3. Suction coefficient K_s as a function of total P and open P_o porosity of samples from porcelain (*1*) and faience (*2*) pastes before (*a*) and after firing at 1200°C (*b*).

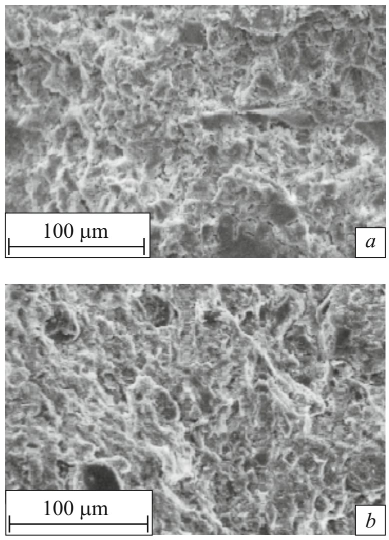

Fig. 4. Microstructure of porcelain (*a*) and faience (*b*) samples fired at 1200°C.

of slabs, with the minimum inhomogeneity coefficient, were the most homogeneous with respect to open porosity. Samples in the shape of cylinders with a recess had the least homogeneous surface, i.e., the surface inhomogeneity of the samples increased as the shape of the article became more complex.

The degree of inhomogeneity of the samples, determined with the open porosity of the surface, decreased with a decrease in the shape factor for samples of the same shape but different molding method, and this is in agreement with the results of determining the homogeneity of the samples based on markers [4]. With respect to the decrease in the degree of inhomogeneity of the surface of the samples, the molding methods can be placed in the following order: plastic molding, semidry molding for the slab samples, and slip casting for the cylindrical samples.

In examining the effect of the molding method on the degree of surface inhomogeneity of samples of different shape, we note that the most homogeneous surface can be obtained on the ends and sides forming the cylinder by using casting in gypsum molds. The samples obtained by semidry molding were characterized by greater surface inhomogeneity both on the ends and on the surfaces forming the cylinders (Fig. 5*a*). Greater inhomogeneity of the base surface of the slabs made by slip casting was also found (Fig. 5*b*). The cylinders with a recess formed by semidry molding were distinguished by the most inhomogeneous surface, while the samples obtained by slip casting had the lowest inhomogeneity coefficient (Fig. 5*c*).

The samples made from faience containing a large amount of finely disperse clay fraction had the most homogeneous open porosity regardless of their shape (see Fig. 5). The samples made from a mixture of porcelain paste and a filler had the most inhomogeneous surface porosity regardless of the molding method.

According to the data obtained in studying the samples, the homogeneity of their surface was higher before firing than after firing, regardless of the shape and the material, as well as the molding method. The samples from semidry and plastic molding fired at 1100°C exhibited a sharp increase in the surface inhomogeneity coefficient. The degree of inhomogeneity decreased with an increase in the firing temperature (Fig. 6). However, for the samples formed by casting into gypsum molds and then fired at dif-

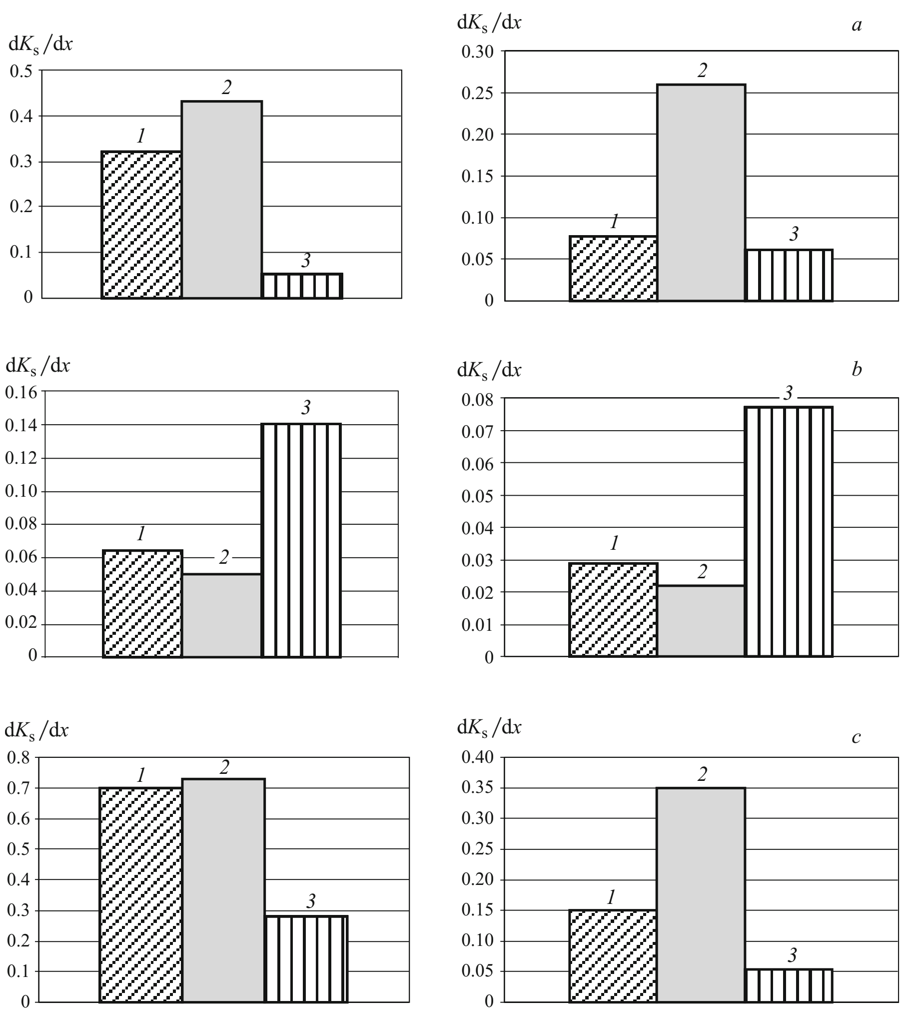

Fig. 5. Inhomogeneity coefficient of samples of porcelain (on left) and faience (on right) in the shape of a cylinder (*a*), slab (*b*), and cylinder with a semicircular recess (*c*) fired at 1000°C: *1*) plastic molding; *2*) semidry molding; *3*) slip casting.

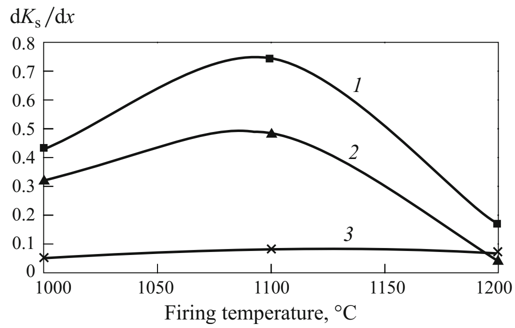

Fig. 6. Change in the inhomogeneity coefficient of samples of porcelain in the shape of a cylinder with an increase in the firing temperature: *1*) semidry molding; *2*) plastic molding; *3*) slip casting.

ferent temperatures, the inhomogeneity coefficient varied insignificantly.

It was thus confirmed that the molding method is the determining factor in production of articles with homogeneous surface characteristics.

The effect of a change in the structure of the samples after firing at different temperatures on the degree of their homogeneity, evaluated with the porosity of individual surface sections, was established.

The capillary suction method can be used to assess the surface inhomogeneity of articles of different structure and shape made by different molding methods without destroying the article and without using complicated and expensive equipment. The method can be used for fast evaluation of water suction, which is often a characteristic of the degree of sinterability of ceramics.

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